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(54) Title: **COMPONENT PRODUCED OR PROCESSED BY POWDER METALLURGY, AND PROCESS FOR PRODUCING IT**

(57) Abstract: The invention relates to components which are produced or processed by powder metallurgy, and to processes for producing components of this type. The components produced by powder metallurgy are intended both to have porous regions and to provide fluid-tight properties, and it should also be possible to produce them at correspondingly low cost and suitably flexibly. For this purpose, a component of this type has at least one porous region, which is formed from an intermetallic phase or solid solutions. However, it may also have a corresponding surface coating. Moreover, in a component of this type there is at least one areal fluid-tight region which is formed from a metal or metal alloy of the corresponding intermetallic phase or solid solution.

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Component produced or processed by powder metallurgy,
and process for producing it

5 The invention relates to components which are produced
by powder metallurgy or alternatively are processed by
powder metallurgy and have at least one porous region,
which is formed from an intermetallic phase or solid
10 solutions, or have a surface coating of this type. In
addition, the invention also relates to corresponding
production processes. In this context, the term
processing by powder metallurgy is to be understood as
meaning a corresponding, retrospective processing of
15 semifinished products, such as for example metal foam
structures, by powder metallurgy.

The prior art has disclosed possible ways of producing
sintered porous bodies which have been formed from
intermetallic phases or solid solutions. A process of
20 this type is described, for example, in DE 101 50 948.
In this document, it is proposed for a powder with a
sintering activity which at least forms intermetallic
phases or solid solutions to be applied to the surface
of a porous base body. Then, the formation of
25 intermetallic phases or solid solutions is supposed to
be initiated by means of a heat treatment. At the same
time, the surface area can thereby be increased.

Although the bodies produced in this way have a
30 relatively low inherent mass and also, if suitable
intermetallic phases or solid solutions are selected, a
high thermal stability, they cannot readily be used for
some applications. This is true in particular with
regard to use as a sealing element without additional
35 assembly or connection to components which are
impervious to the various fluids.

Therefore, it is an object of the invention to provide
components which are produced by powder metallurgy and

have both porous regions and fluid-tight properties and which can also be produced flexibly and at low cost.

According to the invention, this object is achieved by components which have the features of claim 1. Advantageous production processes result in accordance with claims 10, 13 and 14. Advantageous configurations and refinements of the invention can be achieved by the features listed in the subclaims.

10

The component according to the invention which is produced by powder metallurgy or is additionally processed in this way accordingly includes at least one porous region, which is formed from an intermetallic phase or solid solutions. However, a porous region of this type may also be provided with a corresponding surface coating which is formed from an intermetallic phase or solid solutions of this type.

20 Furthermore, there is at least one areal fluid-tight region which is formed from a metal, a metal alloy of the corresponding intermetallic phase or the corresponding solid solution.

25 The term fluid-tight is to be understood as meaning at least imperviousness to certain liquids, but also, under certain circumstances, gas-tightness and even imperviousness to low-molecular gases or gases with a low atomic number.

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In an advantageous configuration, the fluid-tight region may form part of the outer shell of the component, which the correspondingly porous region may then adjoin in one direction.

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However, it is also possible for a fluid-tight region of this type to be surrounded by the porous region. In this case, the fluid-tight region may form a type of core or alternatively a barrier within a component.

Nickel, aluminum, molybdenum, tungsten, iron, titanium, cobalt, copper, silicon, cerium, tantalum, niobium, tin, zinc or bismuth can be used to form the intermetallic phases or solid solutions. It has proven particularly advantageous for at least the porous region to be made from nickel aluminide or to use a corresponding surface coating made from nickel aluminide, since this also makes it possible to achieve very good thermal stabilities.

However, the porous region may advantageously also be formed in such a way that a porosity changes in the direction of the areal, fluid-tight region. This may be effected in steps, i.e. in layers with different porosities within the individual layers, or a continuously graduated form.

The fluid-tight region should advantageously have a density which is over 96% of the corresponding theoretical density.

In one embodiment, however, the fluid-tight region may be formed from a pure metal or a metal alloy of the corresponding intermetallic phases or of a solid solution which is formed areally, for example in the form of a plate. For example, a porous region can be arranged on a nickel component which is, for example, of plate-like design and a porous region, which either consists of nickel aluminide or is surface-coated with nickel aluminide, can be joined by material-to-material bonding to it, as described in more detail below.

Furthermore, it is possible for at least one passage or an aperture to be formed within the fluid-tight region. A passage can be used, for example, for liquid or gaseous coolant to pass through. However, it is also possible to use a passage of this type and adjoining openings to generate a reduced pressure all the way

into the porous region, so that a sucking or vacuum action can be achieved in that region.

5 However, apertures can also be used to secure a component according to the invention using mechanical means.

There are a number of alternative options for producing and/or coating components according to the invention.

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For example, to produce components of this type, it may be expedient to use different starting powders. In this case, a starting powder which has a sintering activity and forms intermetallic phases or solid solutions
15 should be used at least to form an areal, fluid-tight region. This makes it possible to make use of the effect whereby an increase in volume is observed during sintering, causing sufficiently dense sintering of the corresponding region, so that the required fluid-
20 tightness can be achieved.

Starting powders with a mean grain size $d_{50} < 50\mu\text{m}$ should be used in particular to form the porous region during sintering, it being possible, for example, to
25 form the stepped or graduated porous regions which have already been mentioned above to be formed by means of a suitable selection of different grain size fractions.

However, it is also possible, in order to produce
30 components according to the invention, to produce starting powders of the abovementioned grain size fraction in combination with a powder which has a sintering activity and is obtained by high-energy milling.

35

For example, a porous region may be formed exclusively from a starting powder of this type, while an adjoining region, which is likewise porous, may be formed by means of a mixture of this starting powder with a

powder which has a sintering activity and is obtained by high-energy milling, and for a fluid-tight region then to be formed exclusively by means of a starting powder which has a sintering activity and is obtained
5 by high-energy milling.

These different powders employed have different properties during the sintering. In this context, in particular the differing shrinkage is of importance.

10

For example, a powder preform which has been prepared for the powder metallurgy production of components according to the invention may have locally differing dimensions which take account of the different starting
15 powders and their shrinkages which are observed during sintering, so that after sintering a component which is at least near net shape can be provided, requiring at most only slight remachining.

20 During production of a powder preform of this type, by way of example regions in which the powder preform contains starting powders with a higher sintering activity, such as for example powder mixtures obtained by high-energy milling, or have been formed in such
25 regions exclusively from powders of this type with corresponding binders, are characterized by higher shrinkages, which have to be taken into account accordingly.

30 In another alternative, however, it is also possible for components according to the invention to be produced in such a way that a porous structure which is to form the porous region has already been areally coated with a powder which has a sintering activity and
35 forms intermetallic phases or solid solutions. Then, the coated region can be formed in a fluid-tight manner on the corresponding surface of the components by means of a sintering operation.

In this case, by way of example, it is possible to use a porous starting structure such as a semifinished product, comprising a corresponding intermetallic phase or a solid solution.

5

However, it is also possible for a porous structure, likewise in the form of a semifinished product, such as a metal foam, preferably a nickel foam, to be surface-coated with a powder which forms intermetallic phases or solid solutions, as is known from DE 101 50 948, and for an areal layer then additionally to be formed on a surface from a powder which has a sintering activity and forms intermetallic phases or solid solutions and which then likewise forms the fluid-tight region during sintering. For example, the porous structure, i.e. the porous region of a component according to the invention, can be correspondingly modified and the fluid-tight region formed in a sintering operation.

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20 A further alternative production option consists in a metallic element, which is areal and fluid-tight at least in regions and is to form the fluid-tight region, to be joined to a porous structure, which then forms the porous region, by material-to-material bonding.

25 This can be achieved by means of a sintering operation in which the metallic areal element is coated beforehand with a layer of a powder which contains at least one element of the intermetallic phase or of the corresponding solid solution and forms a material-to-

30 material bond with this powder during sintering. The metallic areal element may likewise be formed from an element of the corresponding intermetallic phase or solid solution or from an alloy of this element.

35 The invention is to be described below by way of example.

Example 1

A starting powder mixture which contains nickel and aluminum was used to produce an example of a component according to the invention. The grain size fraction was in the range between 5 and 30 μm .

A nickel to aluminum atomic ratio of 50/50 atomic % was maintained for the mixture composition. The nickel and aluminum starting powders were mixed with one another for a period of 0.5 h. This mixture M1 was then divided into two partial quantities. One of these partial quantities was subjected to high-energy milling in a Fritsch P5 planetary ball mill at a rotational speed of 250 min/h for a period of 1 h. This resulted in a part mixture M2. In turn, a third part mixture M3 was produced from the mixture M1 and the mixture M2, containing these two mixtures in equal parts.

Components were compacted from these mixtures in advance by die-pressing in the following order: mixture M1, mixture M2 and mixture M3.

Then, a reaction sintering operation was carried out in vacuo at a temperature in the region of 1150°C, and a component according to the invention which has three different porous regions was produced. That part of the component which was formed from powder mixture M3 forms the fluid-tight region, whereas the regions formed from mixtures M1 and M2 had a significantly higher porosity.

It was possible to use the powder mixtures with conventional binders which are known per se and are removed during sintering. The grain sizes of the different starting powders M1 to M3 were kept virtually constant, and accordingly in this example there is no grain size change in the high-energy milling process, only the sintering activity of the powder having been changed.

Example 2

A nickel foam structure is surface-coated with a pure
5 aluminum powder or a nickel-aluminum powder obtained by
high-energy milling. A nickel/aluminum atomic ratio in
the range between 75 to 50 atomic % of nickel to 25 to
50 atomic % of aluminum was maintained. The coating
10 with a powder of this type was carried out in such a
way that an open porosity of the nickel foam was
retained. The nickel foam body prepared in this way was
then coated on one side with a powder M3 as described
in Example 1, after which sintering was again carried
out at a temperature of approx. 1150°C. The
15 corresponding intermetallic phases were formed on the
surface of the nickel foam, and a fluid-type region
comprising nickel aluminide was formed where the powder
M3 was additionally applied.

Patent Claims

1. A component which is produced or processed by powder metallurgy and has at least one porous region, which is formed from an intermetallic phase or solid solutions or has a surface coating of this type, and at least one areal fluid-tight region, which is formed from a metal, a metal alloy, the corresponding intermetallic phase or solid solution.
2. The component as claimed in claim 1, wherein the fluid-tight region forms part of the outer shell of the component.
3. The component as claimed in claim 1, wherein the areal fluid-tight region is surrounded by the porous region.
4. The component as claimed in one of the preceding claims, wherein the corresponding intermetallic phase or the solid solutions are based on nickel, aluminum, molybdenum, tungsten, iron, titanium, cobalt, copper, silicon, cerium, tantalum, niobium, tin, zinc or bismuth.
5. The component as claimed in one of the preceding claims, wherein at least the porous region is formed from nickel aluminide or is coated therewith.
6. The component as claimed in one of the preceding claims, wherein at least the porous region has a porosity and density which change in steps or gradually in the direction of the areal fluid-tight region.
7. The component as claimed in one of the preceding claims, wherein the areal fluid-tight region is formed from a metal or metal alloy of the corresponding intermetallic phase or solid solution.

8. The component as claimed in one of the preceding claims, wherein at least one passage or aperture is formed in the areal fluid-tight region.

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9. The component as claimed in one of the preceding claims, wherein the areal, fluid-tight region has a density of over 96% of the theoretical density.

10 10. A process for producing the component as claimed in claim 1 by powder metallurgy, wherein a starting powder which has a sintering activity and forms intermetallic phases or solid solutions is used to form the areal fluid-tight region.

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11. The process as claimed in claim 10, wherein a starting powder with a grain size $d_{50} < 50\mu\text{m}$ and a powder with a sintering activity obtained by high-energy milling are used for production.

20

12. The process as claimed in claim 11, wherein a powder preform is produced from differentiated starting powders, the dimensions of which preform take account of the different shrinkages of the differentiated starting powders during sintering.

25

13. A process for producing the component as claimed in claim 1, wherein a porous structure, which forms the porous region, is coated with a powder which has a sintering activity and forms intermetallic phases or solid solutions, and the areal fluid-tight region is formed at a surface of the component by a subsequent sintering operation.

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35 14. A process for producing the component as claimed in claim 1, wherein a metallic, areal and fluid-tight element, which forms the fluid-tight region, is coated with a layer of a powder which contains at least one element of the intermetallic phase or solid solution,

and the fluid-tight region is joined to a porous structure, which has been placed on top of the powder layer and forms the porous region, by sintering.

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(54) Title: POWDER METALLURGICAL PRODUCTION OF A COMPONENT HAVING POROUS AND NON POROUS PARTS

(57) Abstract: The invention relates to components which are produced or processed by powder metallurgy, and to processes for producing components of this type. The components produced by powder metallurgy are intended both to have porous regions and to provide fluid-tight properties, and it should also be possible to produce them at correspondingly low cost and suitably flexibly. For this purpose, a component of this type has at least one porous region, which is formed from an intermetallic phase or solid solutions. However, it may also have a corresponding surface coating. Moreover, in a component of this type there is at least one areal fluid-tight region which is formed from a metal or metal alloy of the corresponding intermetallic phase or solid solution.

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A. CLASSIFICATION OF SUBJECT MATTER
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B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
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Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, PAJ, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 01/70436 A (YAHIA L HOCINE ; VICTHOM CORP SOC (CA); JOUIN JACQUES (FR); DAUMAS MAR) 27 September 2001 (2001-09-27) claim 8; example 2	1,3-5,7, 9
X	US 5 634 189 A (ROSSMANN AXEL ET AL) 27 May 1997 (1997-05-27) claims 8,9	1,2,6
X	US 6 241 469 B1 (NAZMY MOHAMED ET AL) 5 June 2001 (2001-06-05) claims 1,2,9	1,8
X	EP 0 657 950 A (KATAYAMA TOKUSHU KOGYO KK) 14 June 1995 (1995-06-14) column 11, line 13 - line 44; claim 2	1,10,11
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☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 2002/172836 A1 (TAKAYAMA TAKEMORI ET AL) 21 November 2002 (2002-11-21) paragraphs '0037!, '0041!, '0042!, '0049!; claim 13	1,14
X	US 4 855 101 A (MOHS RUDOLF ET AL) 8 August 1989 (1989-08-08) column 2, line 12 - line 24; claims 1,12	1,14
A	WO 01/21347 A (ROCKWELL SCIENCE CT LLC) 29 March 2001 (2001-03-29) page 6, column 34 - page 7, column 15; claim 1	12

Patent document cited in search report		Publication date		Patent family member(s)	Publication date
WO 0170436	A	27-09-2001	FR	2806421 A1	21-09-2001
			AU	4427701 A	03-10-2001
			WO	0170436 A1	27-09-2001
US 5634189	A	27-05-1997	DE	4338457 A1	18-05-1995
			FR	2712218 A1	19-05-1995
			GB	2284825 A ,B	21-06-1995
US 6241469	B1	05-06-2001	DE	19848104 A1	20-04-2000
			DE	59907926 D1	15-01-2004
			EP	0995880 A2	26-04-2000
EP 0657950	A	14-06-1995	JP	3509031 B2	22-03-2004
			JP	7166211 A	27-06-1995
			CA	2136804 A1	11-06-1995
			CN	1109200 A ,B	27-09-1995
			DE	69429153 D1	03-01-2002
			DE	69429153 T2	14-11-2002
			EP	0657950 A1	14-06-1995
			US	5508114 A	16-04-1996
			US	5655295 A	12-08-1997
US 2002172836	A1	21-11-2002	JP	2002180216 A	26-06-2002
			US	2004123698 A1	01-07-2004
US 4855101	A	08-08-1989	DE	3723650 A1	26-01-1989
			EP	0299238 A2	18-01-1989
WO 0121347	A	29-03-2001	US	6517773 B1	11-02-2003
			CA	2382369 A1	29-03-2001
			EP	1216115 A1	26-06-2002
			JP	2004514051 T	13-05-2004
			WO	0121347 A1	29-03-2001